Laboratory Prepared Ash at 750°C and 525°C

1. Introduction

This method is performed in order to obtain the inorganic residue, or ash, from coal or coke sample material. This residue is then used in the digestion methods (EGL Method 12, and EGL Method 22) which prepares samples for ICP-MS and ICP analysis.

2. Interfaces with Other Methods

This method relies on the Calibration of Laboratory Scales and Analytical Balances method (EGL Method 29) and the method for Inorganic Sample Preparation (EGL Method 26).

3. Materials and Equipment

Furnace programmed to ash at both 525°C for 36 hours and 750°C for four hours as well as ceramic crucibles.

4. Procedure

Using the ash percentage calculation from the Ash Percent of Solid Matrices method (EGL Method 01), calculate how much sample material is needed to produce enough ash for each digestion. Weigh out the determined amount into a ceramic crucible. Place the crucible containing the sample in a furnace and select the proper program.

A) For analysis of Major and Minor elements, heat the sample at 750°C for four hours
B) For analysis of Trace elements, heat the sample at 525°C for 36 hours.

After the selected program is complete and furnace temperature is below 100°C, remove crucible and place in a desiccator.

5. Calibration and Quality Control Samples

Check the furnace temperature and program with a thermocouple connected to a data logger at least once annually. Adjust the program/settings control on the front of the furnace to ensure the correct program is selected. Ash lab standards in the same manner as described above.

Duplicate samples are run with every batch to ensure data quality.

6. Limits, Precautions, and Interferences

As a precaution, place the furnace under a hood to help dissipate heat and fumes.

7. Acceptance of Data

To determine if the data generated is acceptable, the duplicate sample ash percent must be within 10% of the original result post combustion. If the weights deviate relatively by more than 10%, the same samples will be re-ashed at the same temperature for the same amount of time and re-weighed. If this does not result in weights which meet the acceptable data criteria, all of the ashed samples will be discarded and new samples will be ashed starting from the beginning of the procedure.

8. Data Handling and Transfer

The weight is transferred from the balance to a Microsoft Excel™ template electronically. The template calculates the ash percent for each sample and the percentages are then transferred into a template which is saved on the shared network drive.

9. References

American Society for Testing and Materials International [ASTM], 2007, Annual book of ASTM standards, section five, petroleum products, lubricants, and fossil fuels, Gaseous fuels; coal and coke: West Conshohocken, Pennsylvania, American Society for Testing and Materials International, v. 05.06, p. 352.

10. Attachments

None.

11. History of Changes

Revision 0: initial issue